



Micro/nano SAXS/WAXS
+
 μ GISAXS experiments at BW4

Small beams for small samples

Jochen S. Gutmann

Institute of Physical Chemistry, Johannes Gutenberg University, Mainz
and
Max Planck Institute for Polymer Research, Mainz



Outline

- Personal motivation: Microcantilever sensors
 - System
 - Microfocus GISAXS
 - Microfocus X-ray reflectometry
- Micro/nano SAXS/WAXS efforts at the HASYLAB
- Sumary

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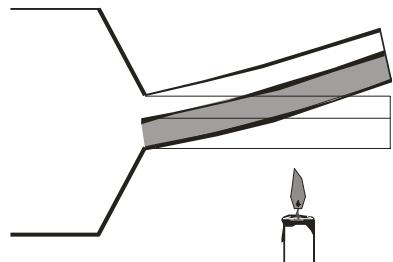


What are micromechanical cantilever sensors?

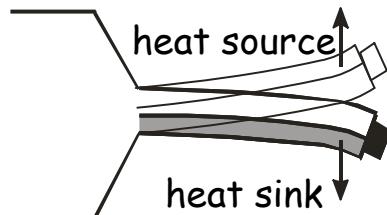


Micromechanical cantilever principle

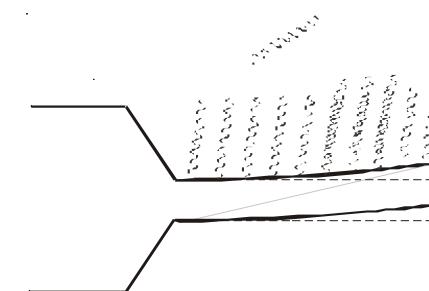
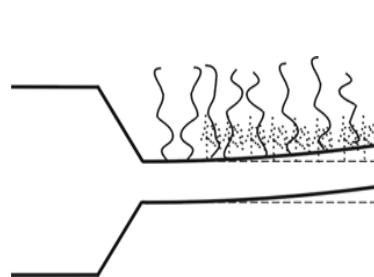
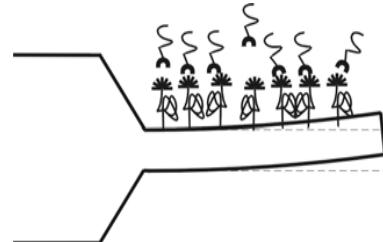
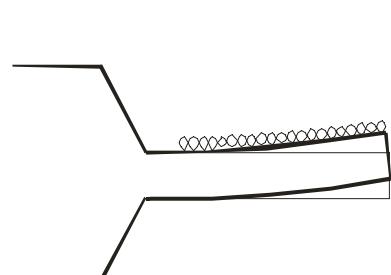
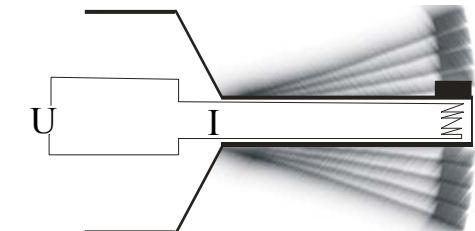
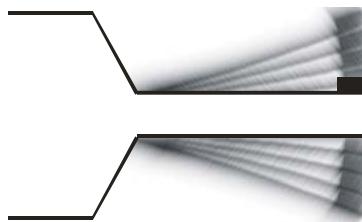
calorimetric detection



static detection



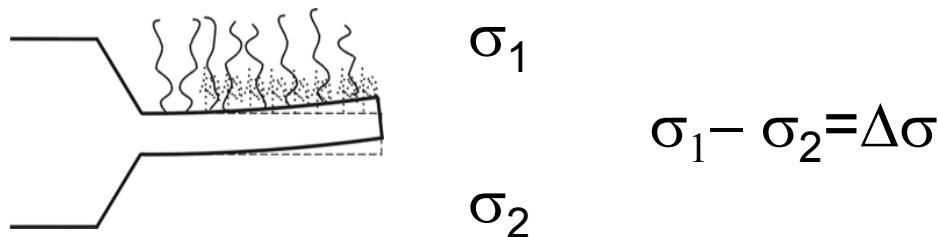
dynamic or gravimetric detection



surface energy changes, steric interaction, electrosatic interaction,
conformational changes, structural phase transitions, stiffness change, volume change

Measurement principle

Differences in surface stress



- static or surface stress detection

$$\Delta\sigma = \frac{Et_s^2}{6R [1-\nu] t_f}$$

$$[\sigma] = N/m = J/m^2$$

$$\frac{1}{R} \approx \frac{3\Delta V}{2L^2} \frac{dz}{dV}$$

- one side coating of microcantilever
- uniform coating
- analysis of bending via Stoney's formula (for constant radius of curvature)



Central problems:

All asymmetrically coated cantilevers are
„bimetallic elements“

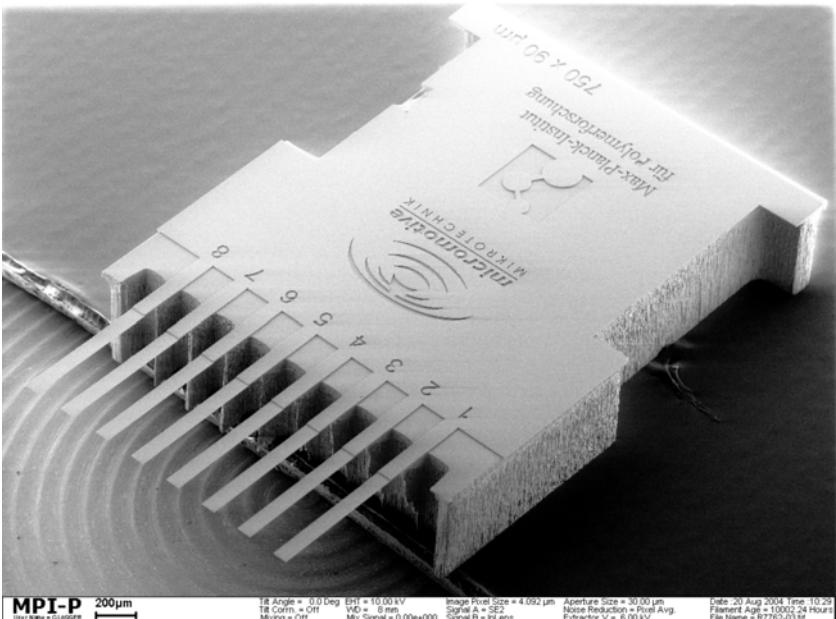
All experiments show drift in the
nm/h range

Solution

Incorporate references into your set-up



Micro cantilever arrays



A 'novel' type of sensor

- Highly sensitive
(pg mass differences)
- Easy to integrate
- Cheap (silicon) technology

But

- Coating may be difficult
- Physisorbed films may be washed off
- Chemical grafting of polymer layer

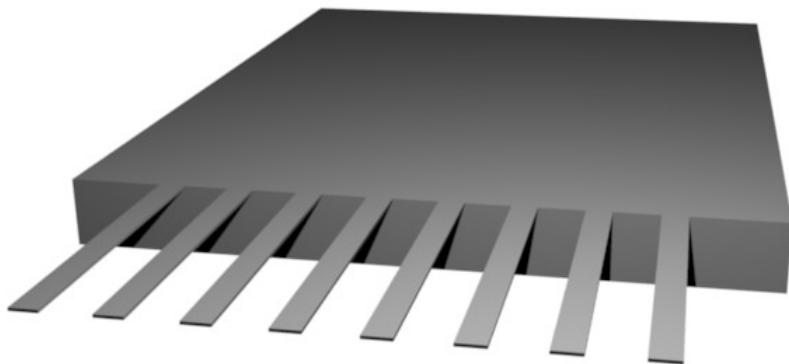


How do we coat only selective cantilever surfaces?

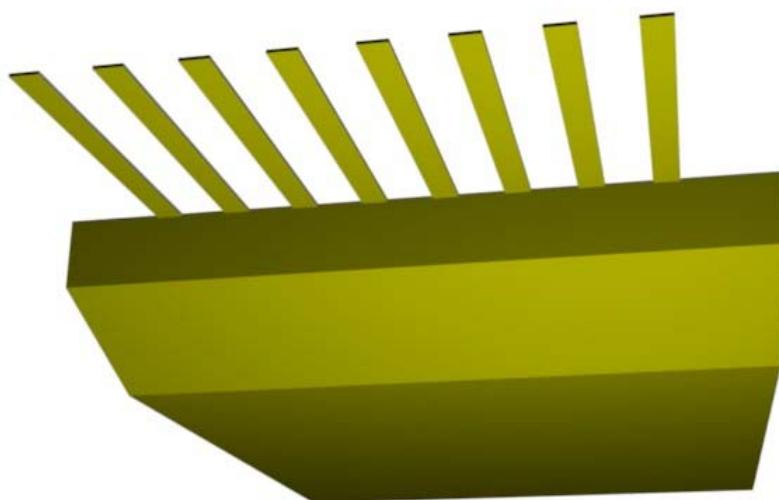
Apply protective masks



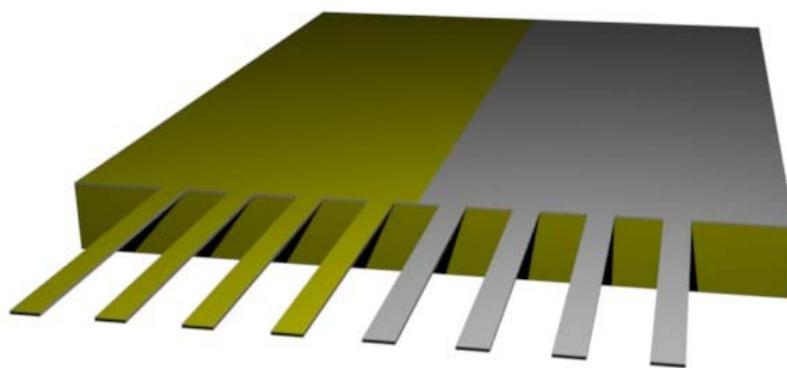
Bare Cantilever



Evaporate gold on bottom

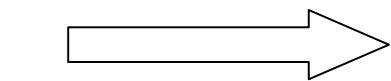
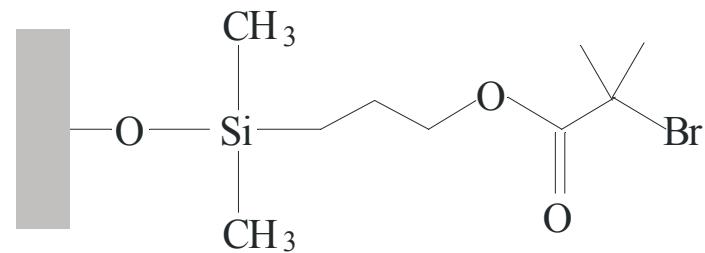
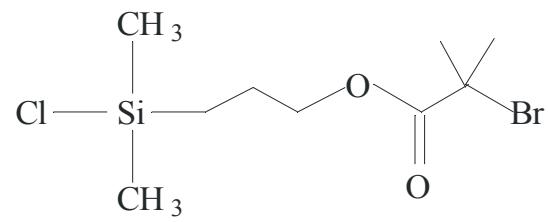


Evaporate gold on half of top

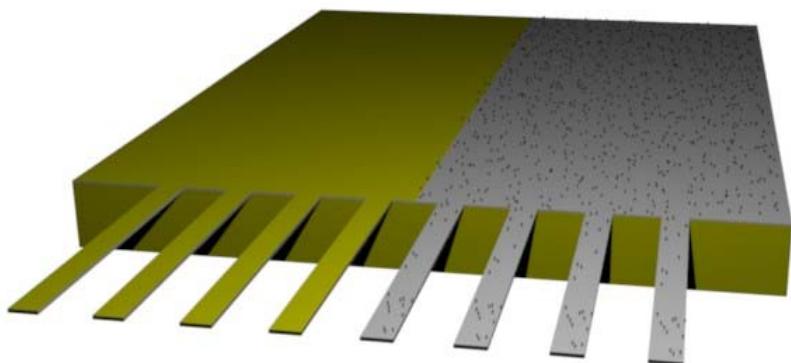


Use a shadow mask to protect half of the cantilevers

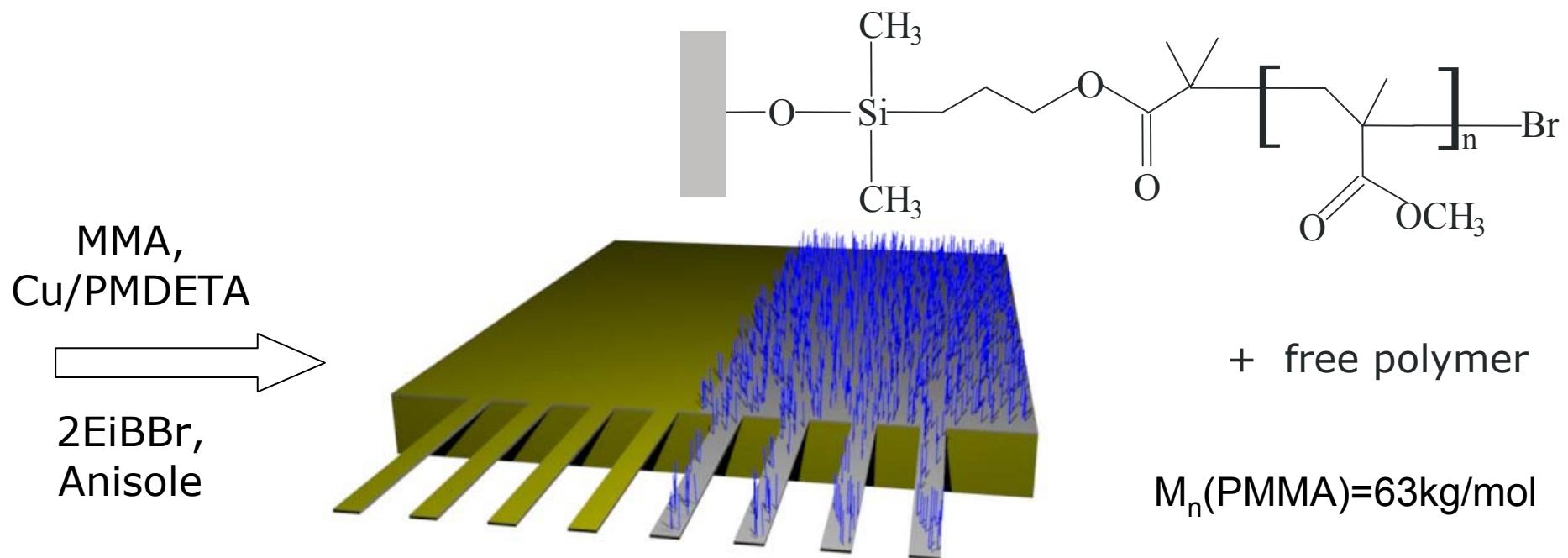
Graft starter for polymerization



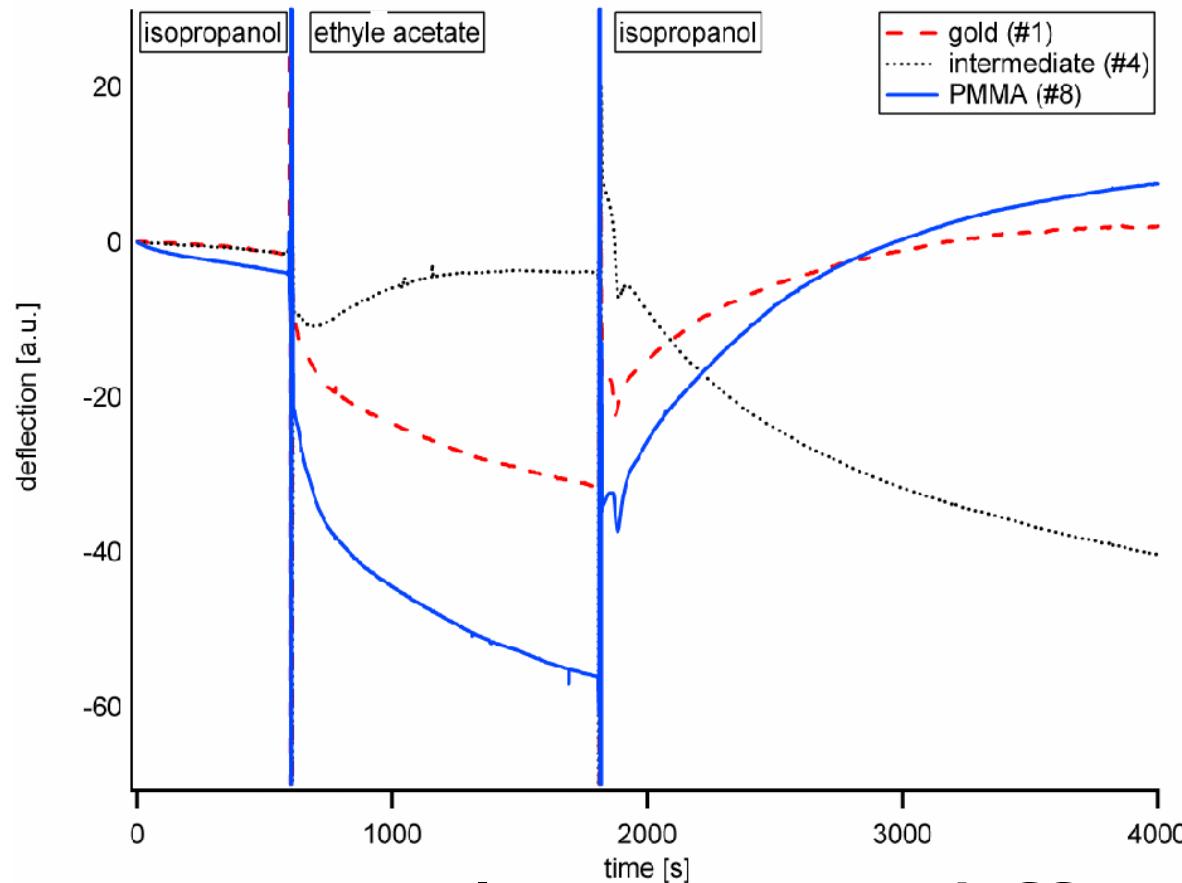
NEt_3 ,
Toluene



Polymer brushes via ATRP of methyl methacrylate

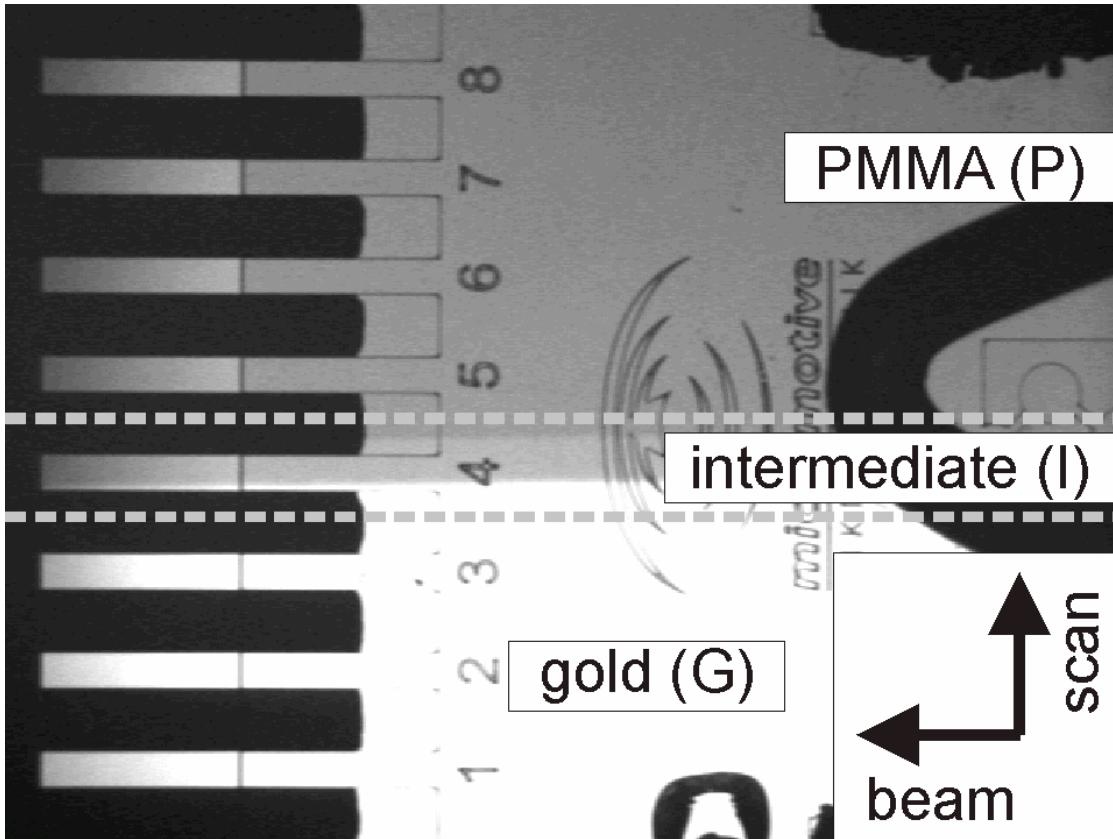


Switching upon exchange of solvent environment



Why is cantilever #4 different?

Optical image



Only partial gold coating for
cantilever #4?

Imaging ellipsometry?
NO: because of gold

AFM imaging?
NO: destructive for cantilever



How do we check layer parameters and mask homogeneity?

Enter microfocus GISAXS



Scattering geometry

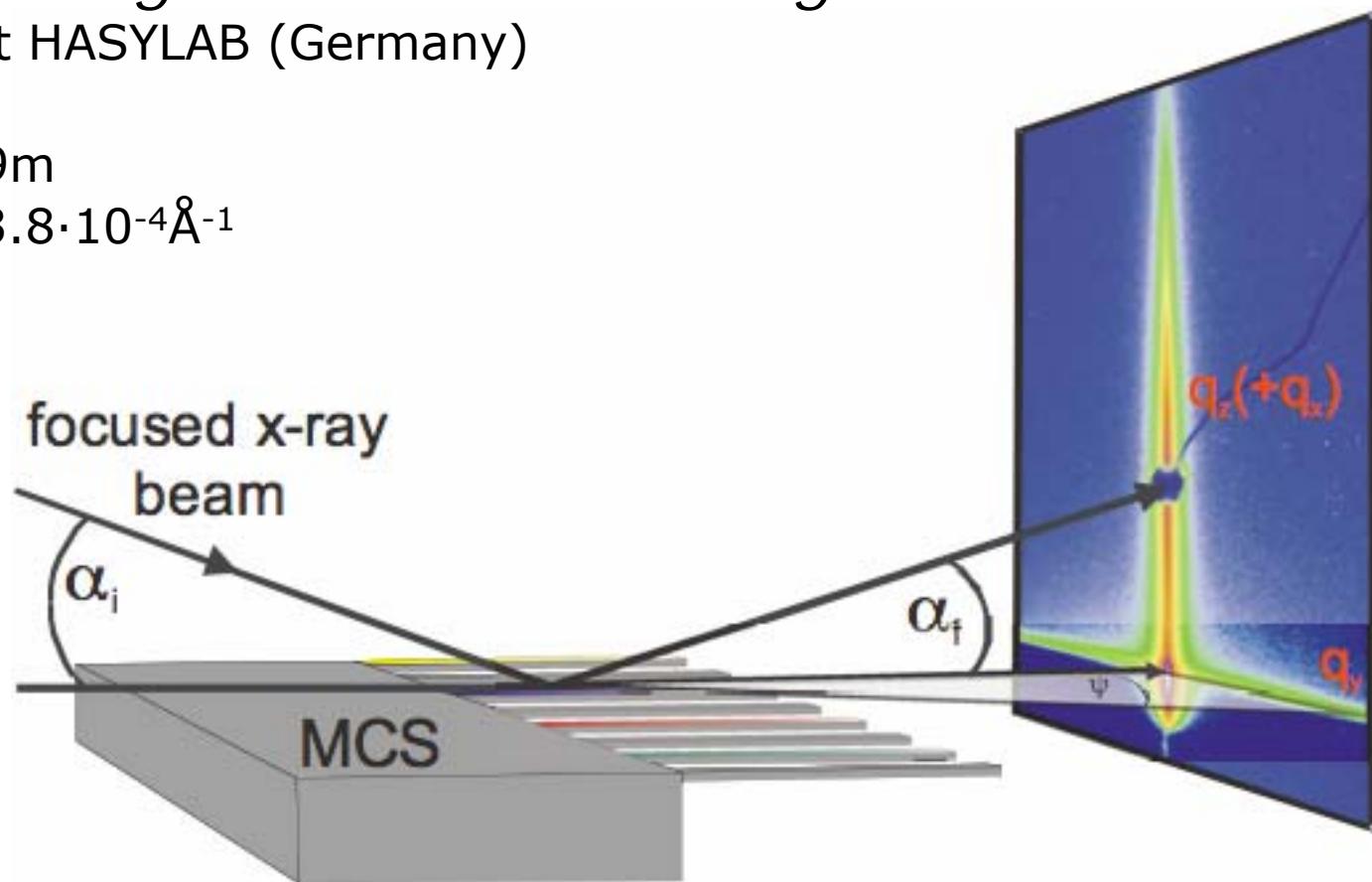
Microfocus Grazing incidence scattering

BW4 beamline at HASYLAB (Germany)

$\lambda = 1.38 \text{ \AA}$

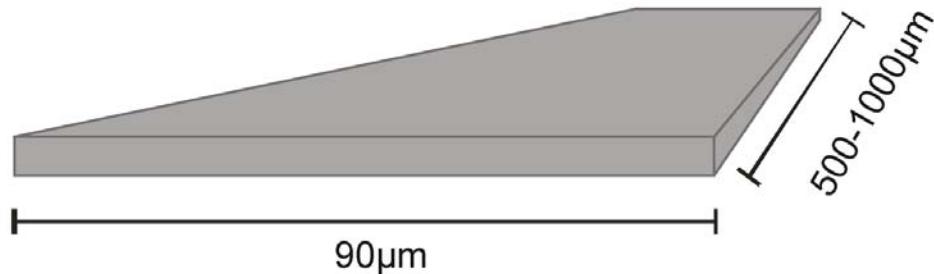
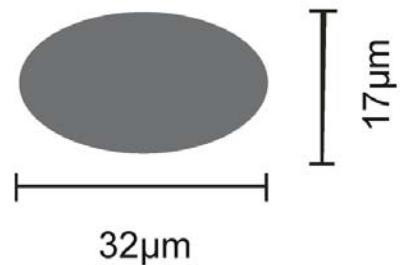
$d_{\text{sample-detector}} \approx 1.9 \text{ m}$

Resolution: $q_y = 3.8 \cdot 10^{-4} \text{ \AA}^{-1}$

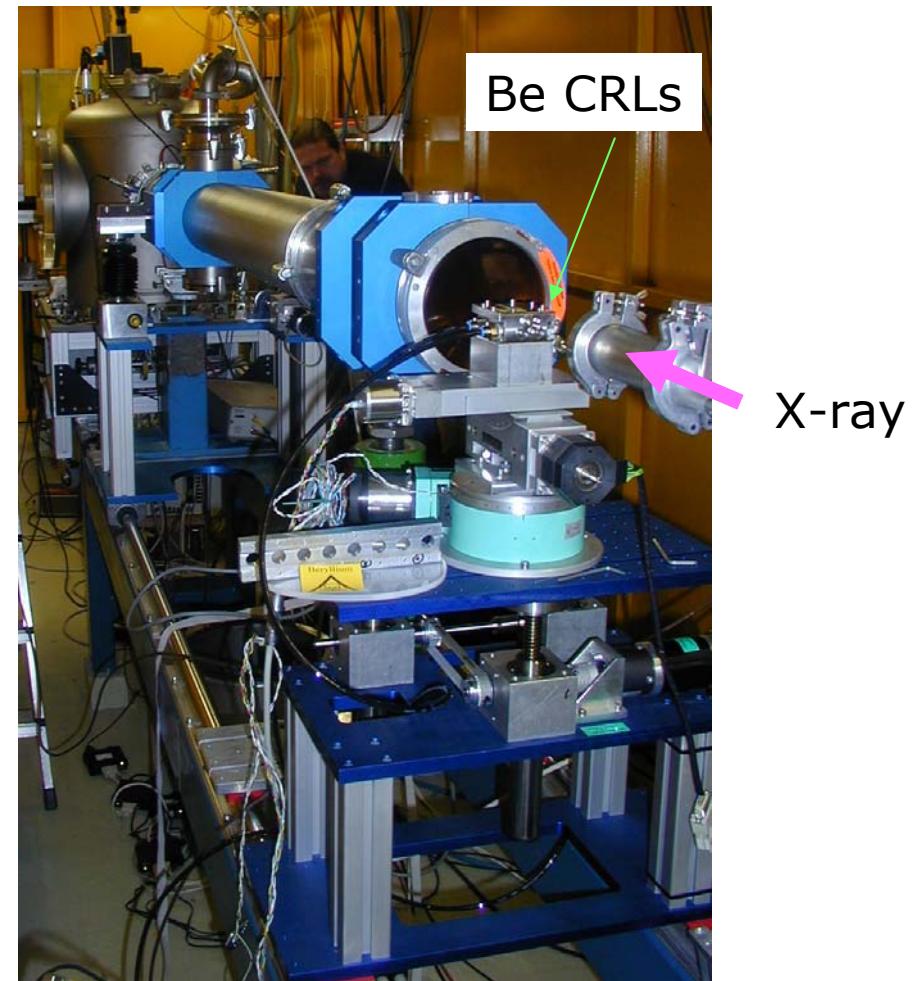


Microfocus with Beryllium CRLs

Beam Dimensions

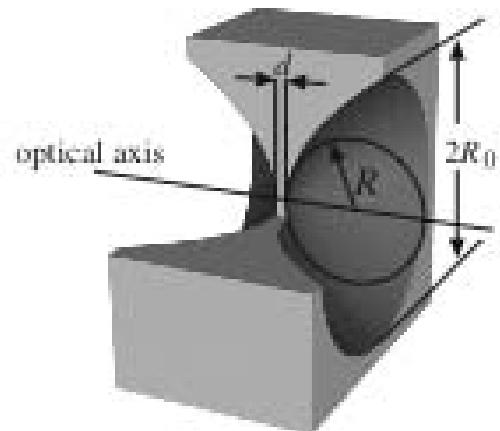


Cantilever Dimensions

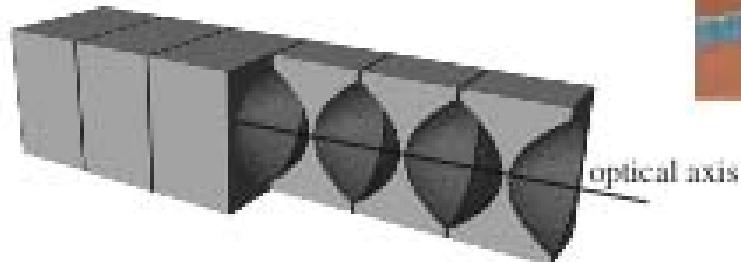


Be CRLs

(a) single lens



(b) compound refractive lens



B. Lengeler, C. Schroer et al.; *J. Phys. D: Appl. Phys.* **38** A218–A222 (2005).

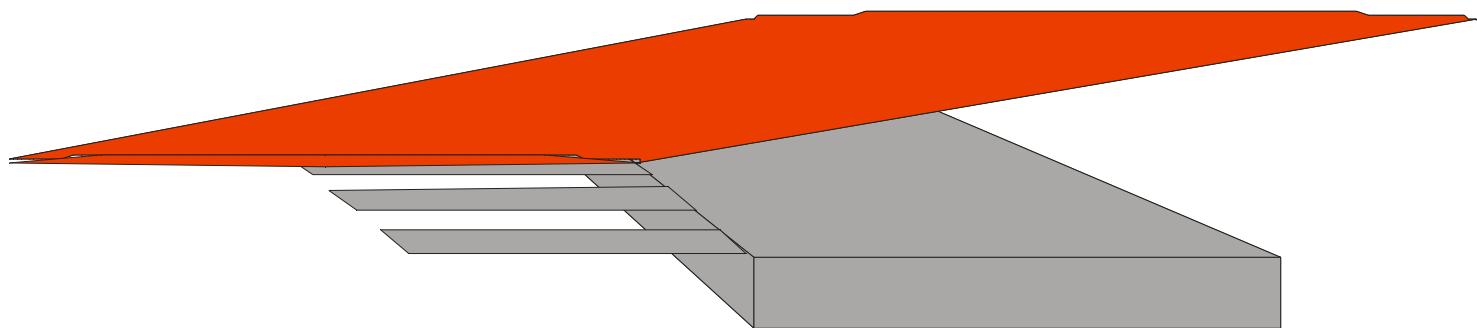
Matching the beam to the cantilever

Problem:

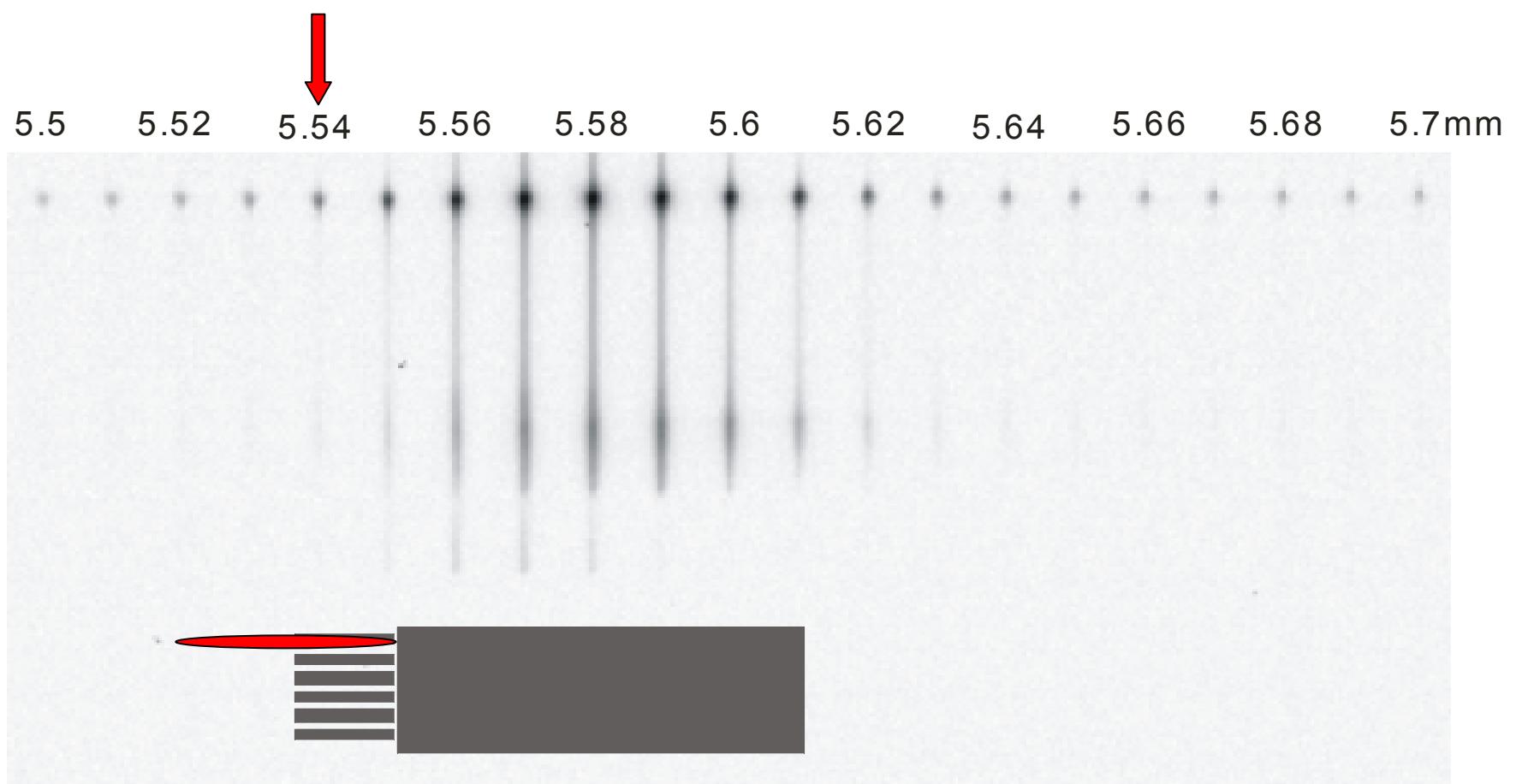
At typical incidence angles we over illuminate by a factor of two.

Solution:

Only use partial beam

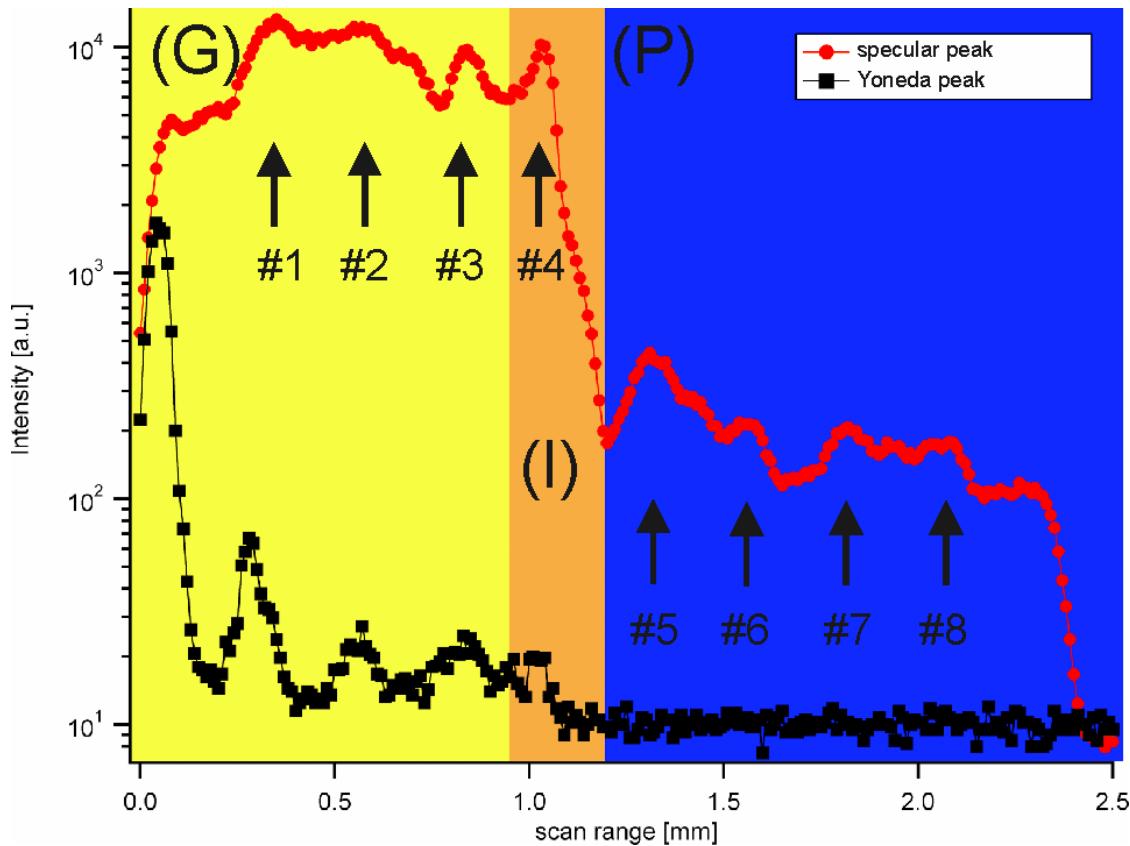


Alignment of cantilever



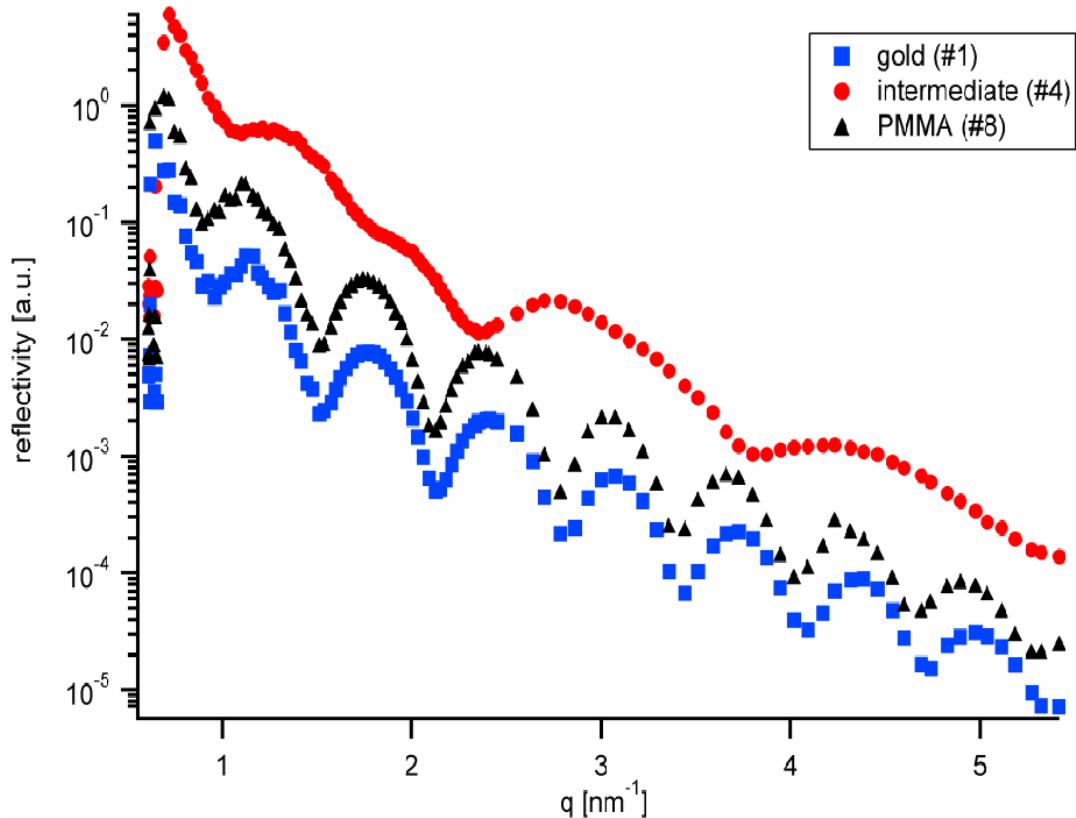
Height alignment in $10\mu\text{m}$ steps

μ -GISAXS scan



⇒ Position dependent gold coverage

Microfocus X-ray reflectivity



$$d_{Au} = 10.3 \text{ nm } (\pm 0.5 \text{ nm})$$

$$d_{PMMA} = 10.4 \text{ nm } (\pm 0.5 \text{ nm})$$

Lack of total reflection edge



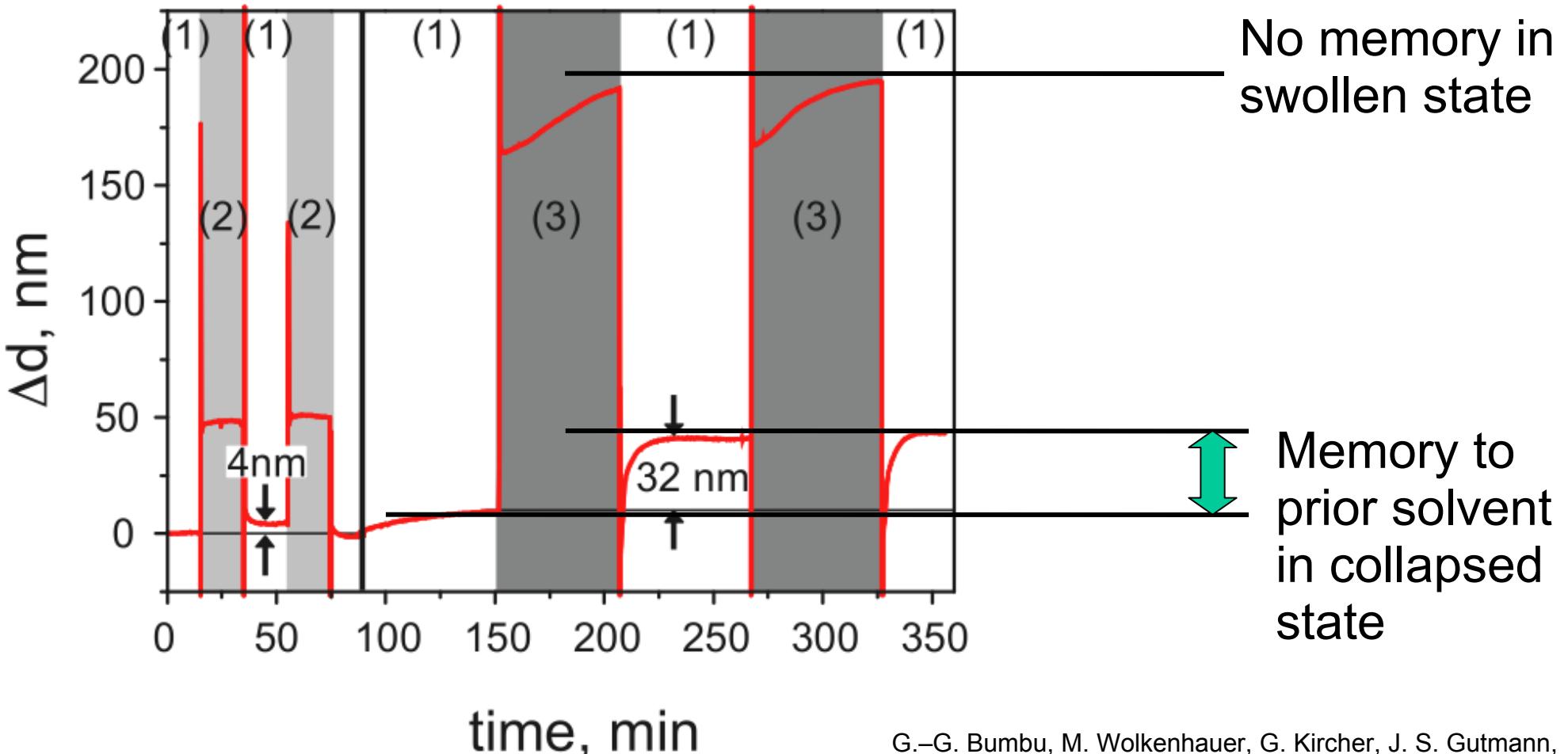
Do brushes remember solvent treatment?

Common finding:
Different initial response after solvent switch

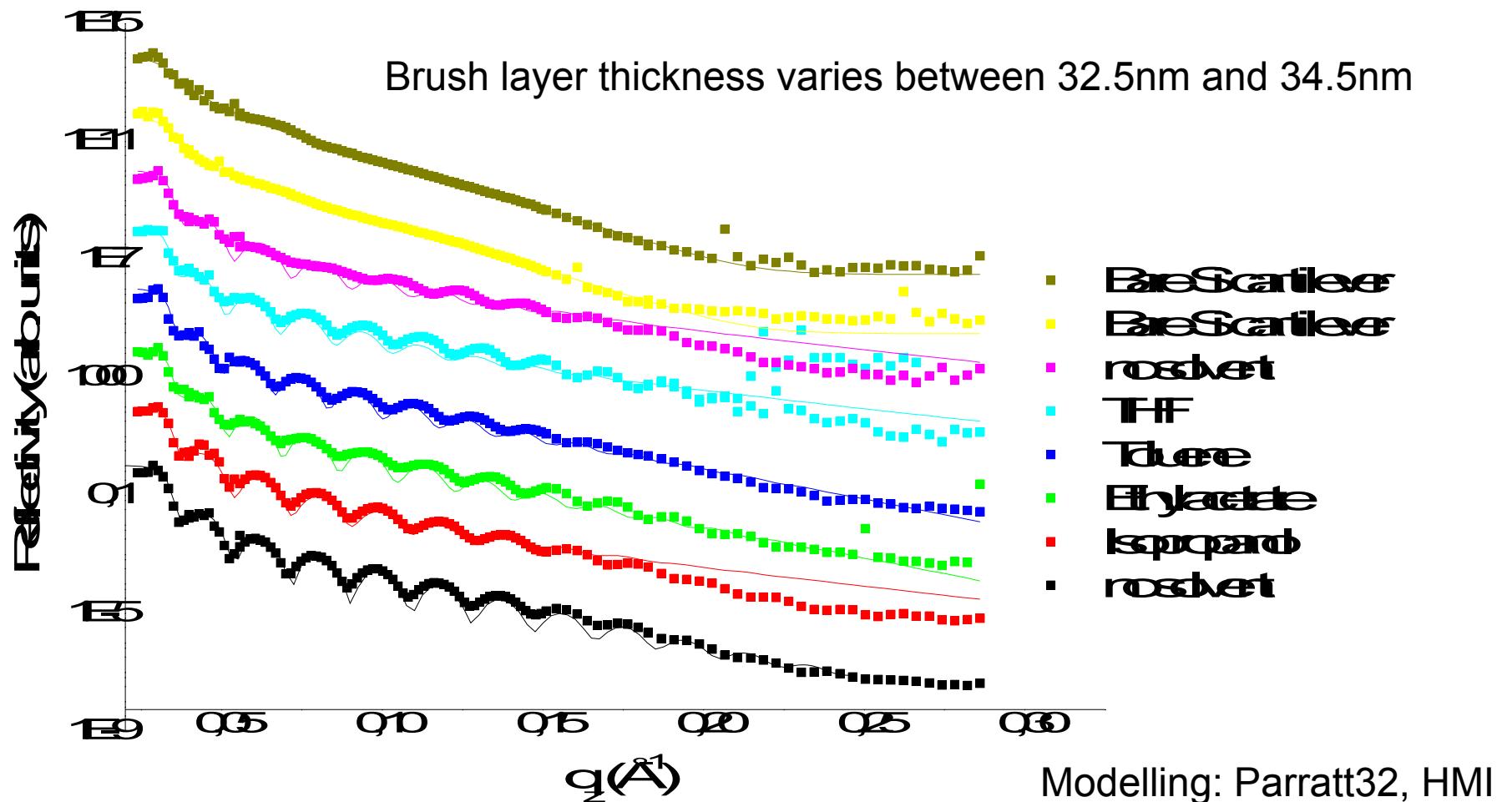


PMMA brushes

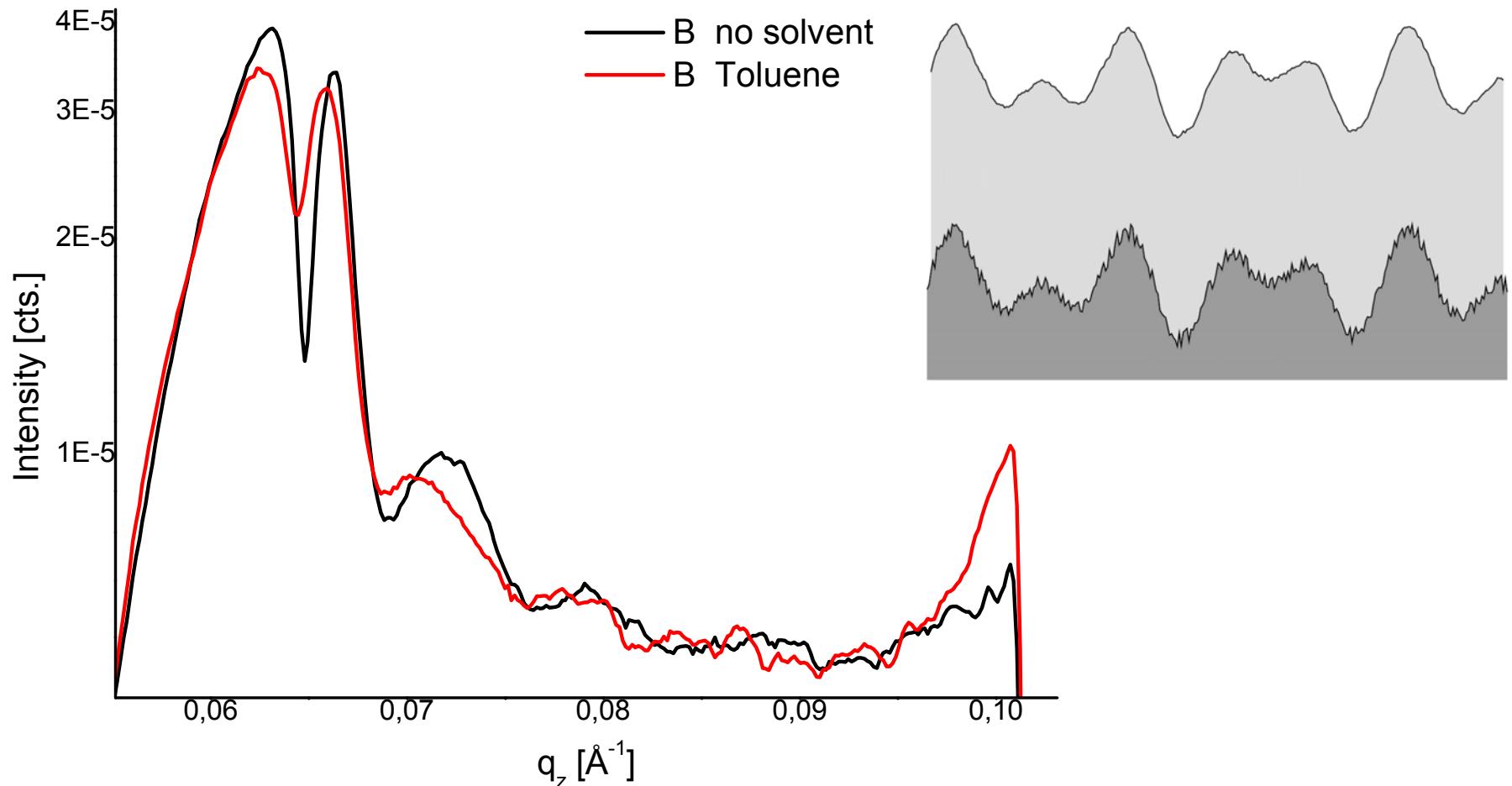
Memory to (prior) solvent treatment



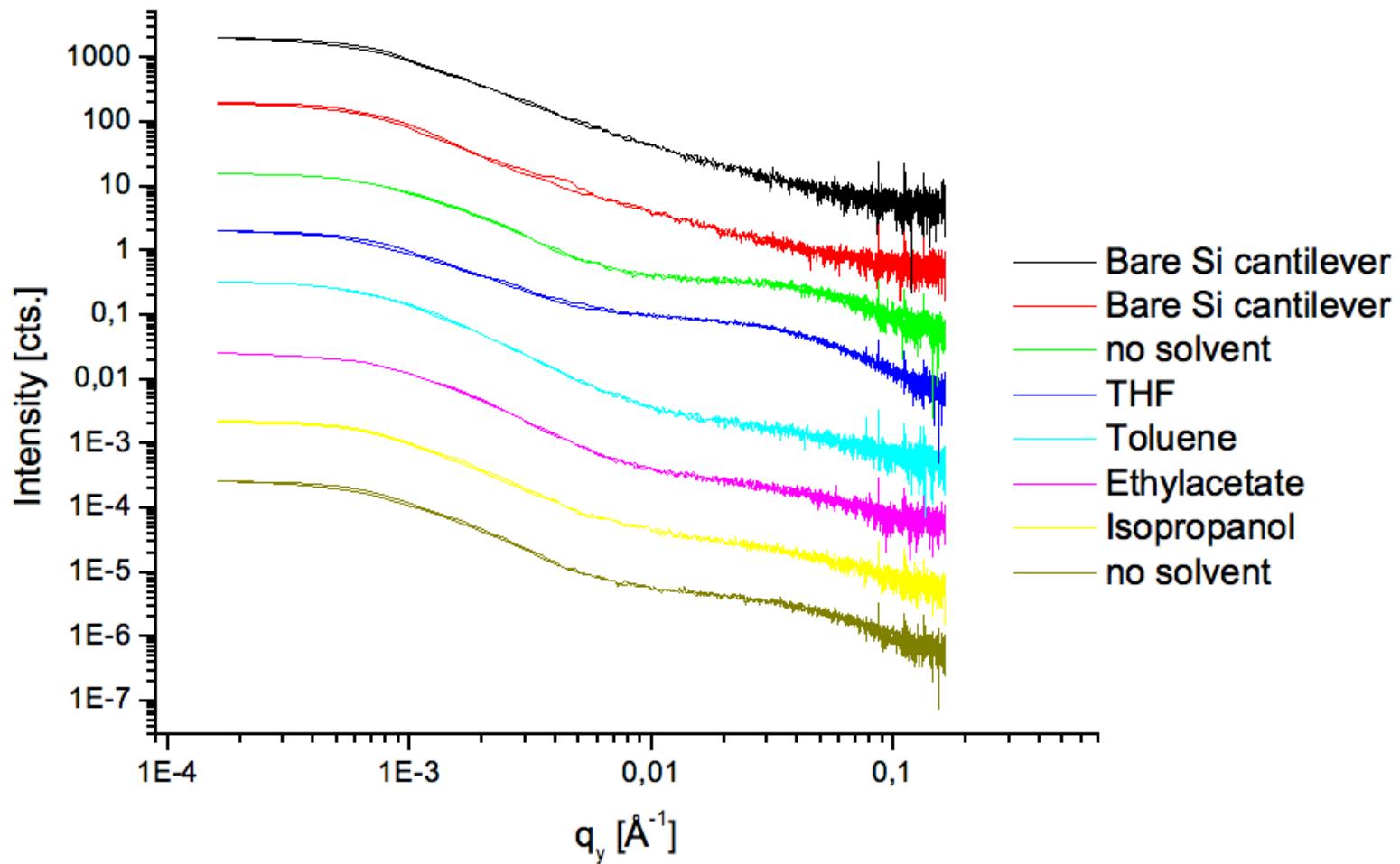
μ -GISAXS investigation of PMMA brush swelling



Evidence for interfacial correlations



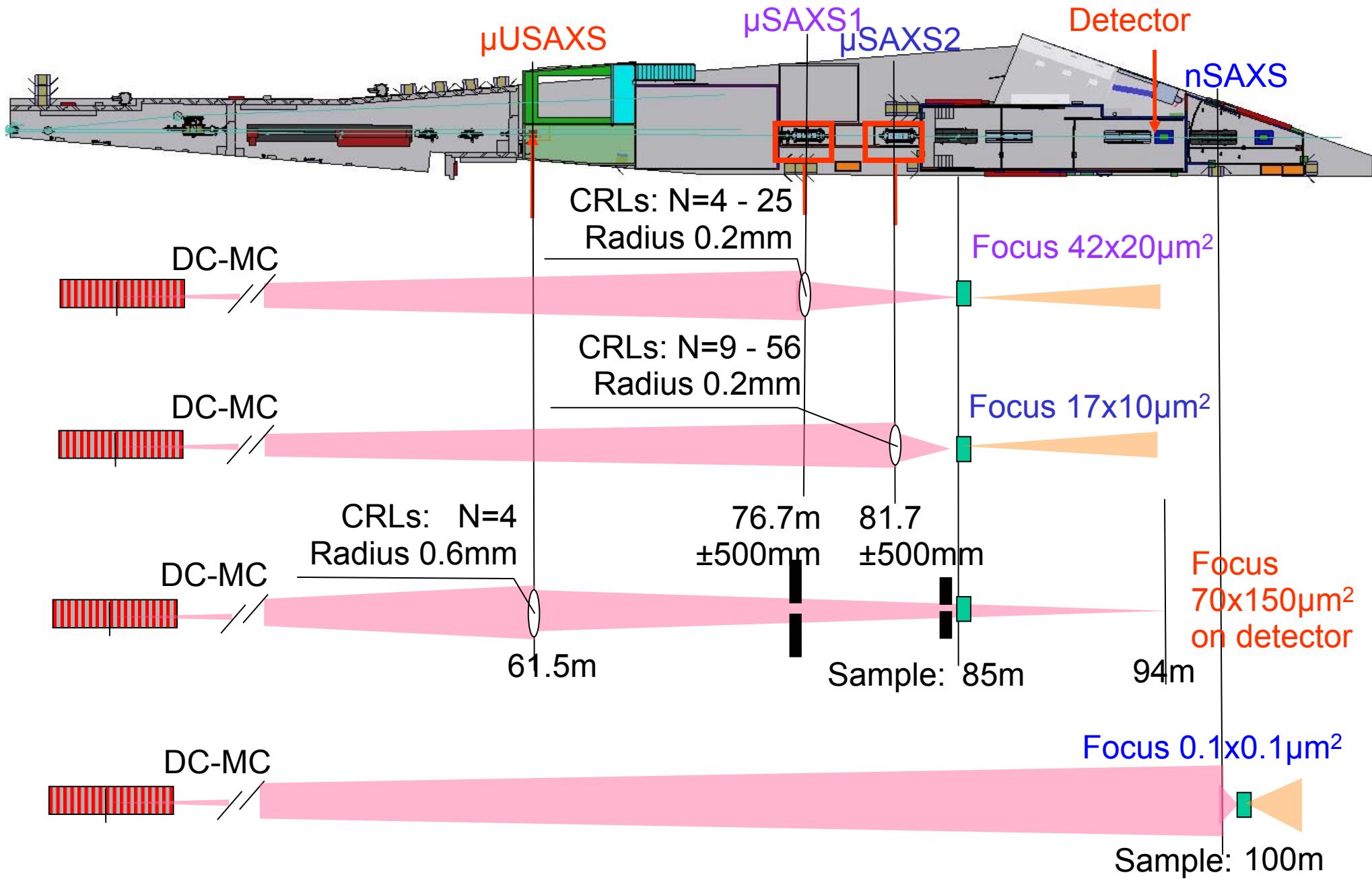
μ GISAXS →Lateral (surface) structures



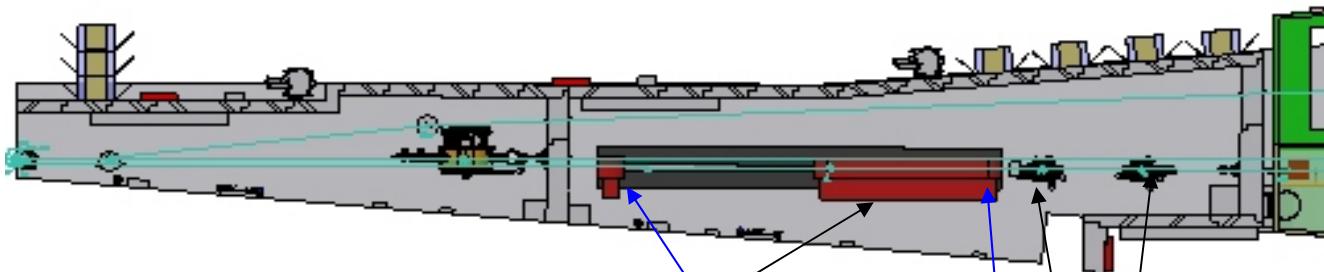
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μ SAXS @ PETRA-III Mission



Design



Sector 2, ID: P03
•U-29, high- β

Large-offset DCM

- Si 111
- vertical offset -490mm
- Fixed exit
- Traverse path: 1300mm
- $8\text{keV} \leq E \leq 23\text{keV}$

Multilayer DCM

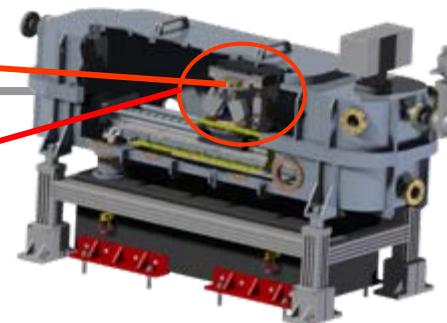
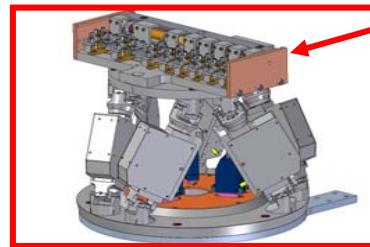
- W/B₄C (10keV), 2nm
- Mo/B₄C (13keV), 1.5nm
- vertical offset -500mm
- Fixed exit, fixed angle

Harmonics suppression Mirror

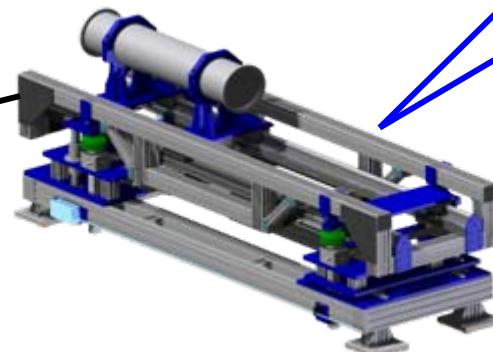
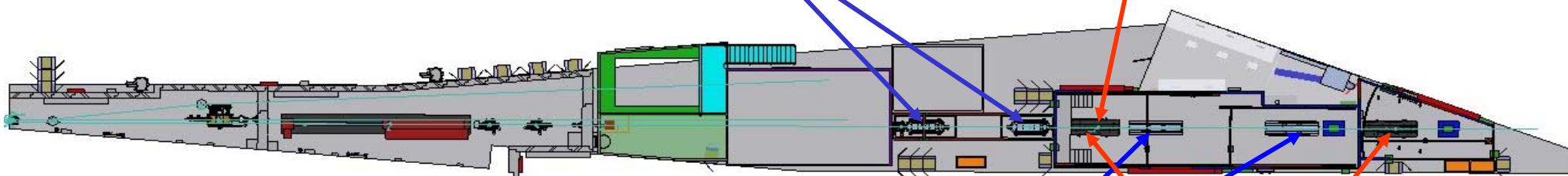
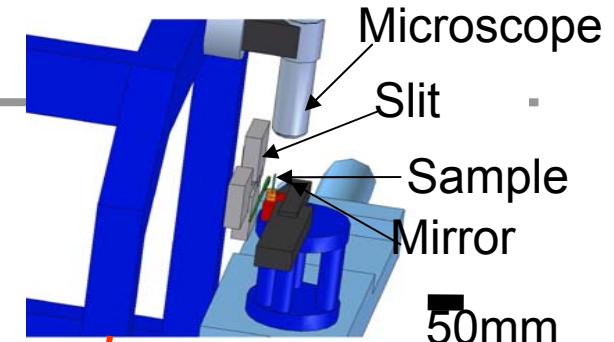
- Quartz substrate
- length: 600mm
- Fixed exit double mirror
- fixed angle: 0.13°
- vertical offset -10mm
- Two additional coating: Pd, Mo
- $8\text{keV} \leq E \leq 23\text{keV}$

Components

Test using 63 BeCRL at BW4

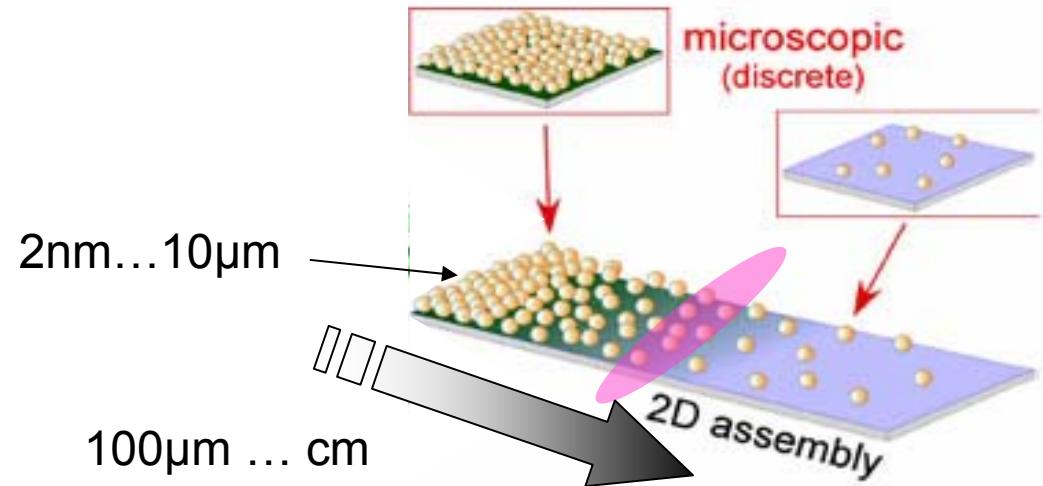
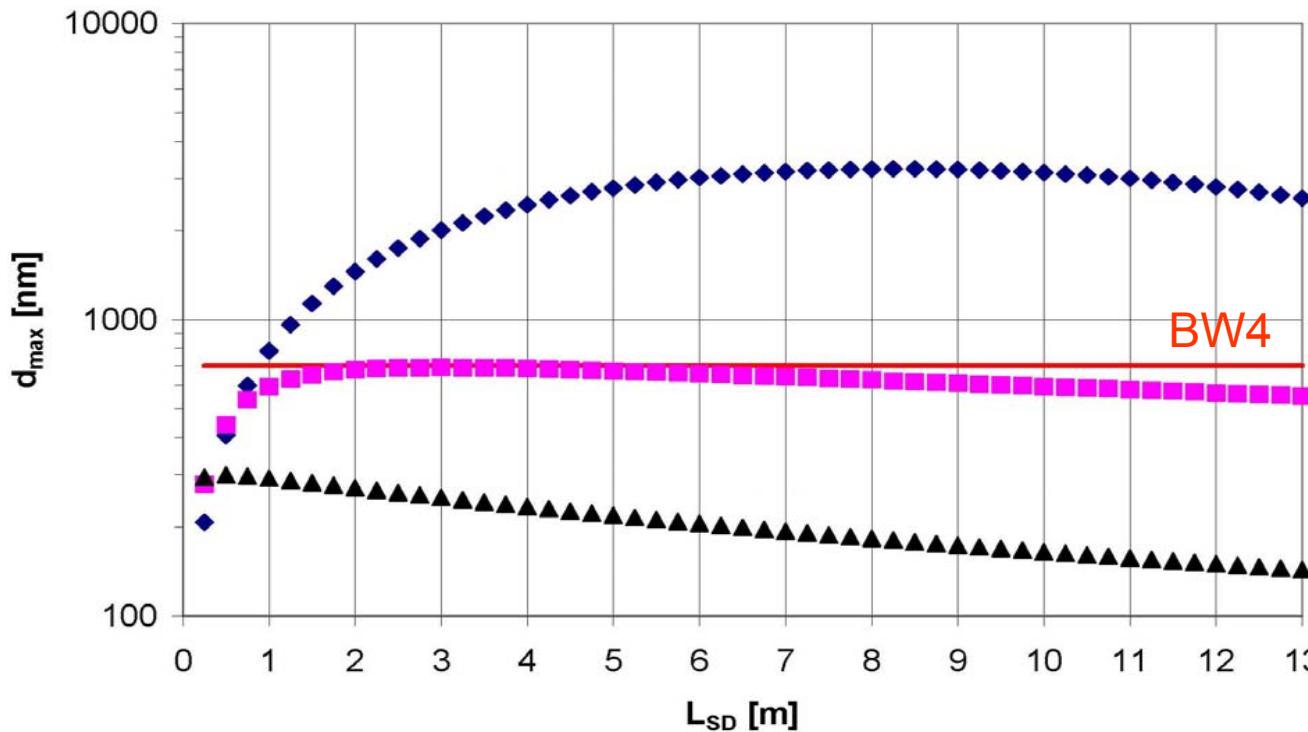


Design underway

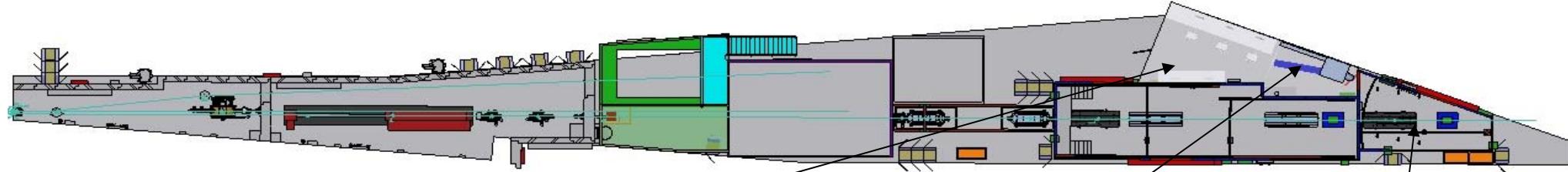


Visit near BW4

Resolution - μ USAXS, μ SAXS1, μ SAXS2



Nanofocus end station EH2



**Control hutch
For EH1 & EH2**

nanofocus EH2

- Nanofocus end station
- SAXS/WAXS
- $B=100\times100\text{nm}^2$

Climate Lock nSAXS

Climate condition:
**hutch operated
independently**

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Summary

μ focus GISAXS

- Scattering on very small samples
- Reflectivity measurement possible
- Off specular scattering possible
→ probe lateral structures and interfacial correlations

Petra - III

- Be CRLs → Microfocus
- Zoneplates → Nanofocus
- μ SAXS, μ WAXS, μ USAXS and μ GISAXS capabilities

Microfocus Extras

- Colinear microscope view
- (Semi-automatic) datatreatment

Thanks to



Dr. Y.-Y. Cheng
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Dr. R. Berger

Dr. S. V. Roth (HASYLAB)

Prof. Dr. H.-J. Butt

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AGFA, IWT

EU-MCTS

DFG

S. Nett
S. Lenz
M. Memesa

Exp. Facilities

HASYLAB



Thank You

and remember.....

cantilever stones



cantilever stone, Glyder Fach (Wales)



...don't deflect

